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(54) Austenitic steel.

(57) A steel grade of austenitic type, extremely resistant particularly to pitting and crevice corrosion, easily weldable, and of high material strength, is characterized by having the following chemical composition in percentages by weight:

max. 0.03	C
0.1–2	Si
8–15	Mn
15–30	Cr
12–20	Ni
3.5–10	Mo
0.35–0.55	N

the balance substantially consisting only of iron, impurities, and accessory elements in normal concentrations.

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AUSTENITIC STEEL

TECHNICAL FIELD

The invention relates to an austenitic steel grade, extremely resistant especially to pitting and crevice corrosion, easily weldable and of high material strength. The invention concerns also the use of the steel for products on which are made exacting demands regarding general corrosion and especially pitting and crevice corrosion, weldability, and strength. The steel is developed specifically for and intended for use within the off-shore industry, e.g. in piping and tanks, and in other environments where the steel is exposed to sea water or to other chloride-containing liquids, such as in thermal power stations where sea water is used for cooling, in the bleacheries of the forest industry, in scrubbers etc. Another field of application is vessels and tubes and heat exchangers for nitric acid, especially those that are cooled by sea water.

BACKGROUND ART

Piping, heat exchangers, tanks and similar equipment and apparatus are mostly made of austenitic steel with 18-21% Ni and over 6% Mo when there is a demand for good weldability, mechanical strength, and very high resistance to pitting and crevice corrosion. The off-shore industry and other industries or plants where sea water is encountered are examples of areas where such demands are made. Other fields of application are within the chemical industry, especially in the chemical bleacheries of the forest industry. There are many corrosion resistant austenitic steel grades that meet very high demands in the respects mentioned, but nevertheless there is a demand for even better materials. At the same time it is desired to cut the cost of materials, for which the cost of such alloys as nickel and molybdenum are very important.

The alloying expenses may be reduced by the use of ferritic ELI-steels, which are also highly resistant to pitting and crevice corro-

sion in sea water and similar environments, provided they contain at least 25% Cr and at least 3.5% Mo. A serious limitation of these materials is that they are not manufactured in thicker dimensions than about 3 mm. If the material is made thicker it becomes brittle and unweldable.

Another practice in order to reduce costs is to replace nickel by manganese partly or wholly in austenitic stainless steel. There are reports that manganese may increase the contribution of chromium to resistance compared to steels with no manganese substitution. The known types of manganese substituted steels are considerably less corrosion resistant, however, than said austenitic and ferritic steel grades, and are no useful alternative to the latter in those environments where the present steel is intended to be used.

The following literature references are intended to illustrate the present state of the art. In the comparative investigations to be presented below reference will be made to data from these references.

Literature references:

(1) Kohl H, Rabensteiner G, Hoehörtler G, VEW: Stainless Steels with High Strength and High Corrosion Resistance. Alloys for the eighties.

(2) Glazkova S A, Shapiro M B: The Resistance of a CrNi-Steel Type 18-12-Mo to Localized Corrosion in Chloride Solutions. Zashch. Metallov 15 (3), May-June 1979, p 320-324.

(3) Bock H E: Korrosionsverhalten eines seewasserbeständigen, nichtmagnetisierbaren CrNiMo-Stahles hoher Festigkeit. Arch. Eisenhüttenwes. 44 (1973)877.

(4) Brigham R J, Tozer E W: Localized Corrosion Resistance of Mn-Substituted Austenitic Stainless Steels: Effect of Mo and Cr. Corr. 32(1976)274.

(5) Letcher B F: An Austenitic Stainless Steel of Improved Strength and Corrosion Resistance (Firth Brown Rex 734). Report from Firth Brown Ltd.

DISCLOSURE OF INVENTION

A primary object of the invention is to provide a steel grade which has the required combination of properties for use in welded constructions in highly corrosive environments, in spite of having a lower, rather than higher, content of expensive alloy metals than comparative conventional austenitic steels. Specifically, an object is to provide a steel with extremely high resistance to pitting and crevice corrosion. A preferred object is to provide a steel of high resistance in other environments also, such as in HNO_3. Typical fields of application are as indicated in the preamble.

A further object of the invention is to provide a steel which is easy to weld with a low energy input without any considerable loss of its corrosion resisting properties in the weld or in the heat-affected zone.

A preferred object is also to provide a steel with a greater mechanical strength than conventional austenitic molybdenum alloyed steels.

These and other objects may be attained by making a steel grade of the following chemical composition in percentages by weight:

Max. 0.03 C
0.1-2 Si
8-15 Mn
15-30 Cr
12-20 Ni
3.5-10 Mo
.35-.55 N

the rest is substantially only iron, impurities, and accessory elements in normal concentrations.

It is desirable to keep the carbon content as low as possible. Normally, the carbon content is therefore maximally 0.02%, preferably maximally 0.015%. The roles of manganese and nitrogen in the steel are complex. The manganese partly functions as an austenitizing agent, partly aids in dissolving nitrogen in the steel. Certain indications suggest that the manganese in this alloy directly influences the corrosion properties favourably. The nitrogen works as an austenitizing agent and adds to the corrosion resistance as well. In order to create a completely austenitic structure, a required amount of nickel is added in addition to manganese and nitrogen. A synergistic effect of molybdenum and nitrogen as regards resistance to pitting and crevice corrosion is also attained with the steel according to the invention. In other words, the nitrogen strengthens the favourable effect of the molybdenum. Chromium is a fundamental element for resistance to general corrosion and also enhances the resistance to other types of corrosion.

A preferred characteristic of the steel is that its so called PRE value (= %Cr + 3.3X % Mo + 16X %N) is at least 41, preferably from 41-45.

It is suitable to include in the steel 14-17 Ni, 9-11 Mn, 18-23 Cr, 4-8 Mo, 0.38-0.48 N and a maximum of 0.015C.

A preferred characteristic of the steel according to the invention is that the chromium equivalent (according to Shaeffler) is at least 24, and the nickel equivalent is at least 25, the ratio of the chromium equivalent to the nickel equivalent being no more than 0.9, preferably no more than 0.8.

It is also important that the steel is non-stabilized, which means that it does not contain any significant, intentional additions of niobium, tantalum, titanium or zirconium. The total amount of these elements must not exceed 0.1 %. Higher amounts would have a too detrimental effect upon the corrosion resistance, because these elements readily combine not only with carbon but also with nitrogen present in the steel to form nitrides, such that the effective nitrogen content would be reduced.

A preferred composition of the steel according to the invention is the following:

Max. 0.015 C
0.2–1.5 Si
9–11 Mn
max. 0.008 S, preferably max. 0.005 S
19–22 Cr
14–17 Ni
4–5 Mo
0.38–0.48 N
Max 0.1 (Nb + Ta + Ti + Zr)
the rest substantially consisting of iron and unavoidable impurities.

Aside from the elements mentioned, copper may be included up to no more than 3%. The possible effects of including copper in the steel according to the invention has not been subject to investigation, however. It is conceivable that it may improve corrosion resistance in strong acids. According to the preferred embodiment, the copper content should be limited to a max. of 0.5%.

Copper at concentrations less than 0.5% is included in the term "accessory elements", signifying such elements as may be found in secondary metal or be traces of process metallurgy additions. Among the former type of elements may be mentioned cobalt. This is an expensive element. Wilful additions of this element should therefore be avoided. Cobalt also has the drawback of becoming radioactive when subjected to radiation, which makes cobalt alloyed material impermissible for such parts of nuclear power stations as are exposed to radiation. The cobalt content therefore should be restricted to a max. of 0.5%. Among traces of elements added for process metallurgy reasons may be mentioned aluminium and calcium. Niobium, vanadium and titanium should not be present at levels exceeding those of impurities.

Further characteristics and advantages of the steel according to the invention will appear from the following account of experiments and investigations carried out.

DESCRIPTION OF EXPERIMENTS AND INVESTIGATIONS CARRIED OUT

The composition of each steel sample made and investigated is presented in Table 1, group 1. Steels Nos. 1-15 were manufactured as melts with a weight of 2 kg. Of these, those with a totally austenitic structure (after rolling and solution heating) were subjected to further investigations, especially regarding their corrosion properties. In order to evaluate the initial investigations, a melt of 50 kg was then manufactured, steel No. 16. Groups 2 and 3 of Table 1 present data for commercially available steels tested, as well as figures from the literature concerning other steels, see the reference list on pp. 2-3.

The B charges (steels Nos. 1-15) were forged to 30 mm square section and rolled to strips of 3 mm thickness and then solution heated ($1100^{\circ}C/1h/H_2O$). No notable problems of poor hot state ductility were encountered.

TABLE 1
Chemical composition (percent by weight, the balance being Fe and accessory elements) of steel samples manufactured and/or investigated.

Group 1: Test samples (B= 2 kg, Q = 50kg, P = 5 tons charges)

Steel No	Charge	C	Si	Mn	P	S	Cr	Ni	Mo	N	Cu	PRE *) value
1	B 1142	.011	.44	4.8	.006	.008	21.9	12.7	3.5	.22		37.0
2	B 1143	.012	.48	4.8	.006	.008	21.9	12.7	2.6	.22		34.0
3	B 1144	.010	.48	9.8	.006	.009	20.0	15.2	3.0	.22		33.4
4	B 1145	.011	.47	9.8	.006	.009	20.0	15.2	4.6	.21		38.56
5	B 1146	.012	.43	7.7	.007	.009	19.9	10.5	4.5	.44		41.75
6	B 1147	.014	.45	9.6	.009	.007	19.8	15.1	4.5	.43		41.5
7	B 1401	.018	.50	1.43	.005	.010	25.2	13.7	1.03	.39	.02	34.8
8	B 1402	.017	.43	1.59	.006	.010	25.9	15.1	2.49	.30	.97	38.9
9	B 1404	.017	.42	1.52	.004	.010	25.1	15.1	1.03	.34	.02	33.9
10	B 1411	.019	1.26	1.55	.006	.010	25.1	13.4	1.04	.24	.02	32.4
11	B 1412	.020	1.27	1.58	.006	.010	24.9	15.1	2.45	.28	.89	37.5
12	B 1413	.020	1.24	1.60	.006	.010	25.0	16.1	3.2	.26	.97	39.7
13	B 1414	.018	1.27	1.58	.007	.010	25.0	16.9	3.9	.23	.95	41.5
14	B 1419	.017	.35	4.66	.005	.010	21.1	15.7	5.5	.38	.95	45.3
		.022	1.20	2.30	.006	.010	24.2	5.6	2.96	.30	1.08	38.8
15	B 1420	.018	.36	1.43	.004	.006	24.1	6.6	2.97	.15	.47	36.4
16	Q 8072	.015	.68	10.3	.011	.007	20.3	15.2	4.5	.48		42.8
35	P62311	.013	.40	10.67	.015	.002	21.3	15.1	4.5	.40	.52	41.2

Group 2: Commercially available steel samples tested

Steel No	Grade	C	Si	Mn	P	S	Cr	Ni	Mo	N	Cu	Rest	PRE*) value
17	AISI 216	.047	.47	8.5	.025	.003	19.7	6.4	2.7	.38	.12		34.7
18	Nitronic 40	.018	.68	9.01	.024	.001	19.93	7.12	.18	.28			25.0
19	" 50	.045	.47	4.81	.025	.012	21.08	13.70	2.28	.28		Nb,V	33.1
20	NU SS 904L	.018	.65	1.67	.027	.004	19.4	24.6	4.4		1.43		33.9
21	NU SS 44LN	.022	.39	1.71	.026	.010	25.1	6.3	1.56	.14	.17		32.5
22	ASTM UNS S 31254	.013	.42	.62	.031	.005	20.3	17.7	5.8	.21	.69		42.8
23	AISI 316L	.014	.43	1.58	.032	.004	18.0	12.7	2.64		.23		26.7
24	NU MONIT	.012	.31	.43	.031	.006	25.3	4.1	3.8		.37		37.8
25	ASTM UNS S 31254	.020	.52	.48	.024	.001	20.1	17.7	5.9	.19		Co	42.6

*PRE value: % Cr + 3.3 x % Mo + 16 x % N

TABLE 1 (contd)

Group 3: Other steel grades as reported in the literature

Steel No	Grade	C	Si	Mn	P	S	Cr	Ni	Mo	N	Cu	Rest	PRE*) value
		max											
26	A 963,ref 1	.03					17.0	16.0	6.3	.15	1.6		40.2
27	A 905, " 1	.04		5.8			25.5	3.7	2.3	.37			39.0
28	A 905, " 1 -weld	.04	.70	4.5			26.0	7.5	2.0	.35			38.2
29	See ref. 2	.02	.36	4.18	.011	.016	18.90	12.10	5.0	.40			41.8
30	" " 3	.03		8.0			20	14	3.1	.40	.14	Nb	36.6
31	" " 4	.04	.5	8.8	.019	.016	20	10.4	4.22	.5	.01		41.9
32	" " 4	.03	.63	5.88	.013	.011	22.1	12.70	3.90	.41			41.5
33	YUS 170 ref. 5	.031	.82	1.59			24.7	13.45	.93	.36			33.5
34	REX 734, ref. 5	.05	.25	4.0			22.0	9.0	2.6	.40		Nb	37.0

*) PRE-value: % Cr + 3.3 x % Mo + 16 x % N

Since the purpose of the invention is to develop a manganese substituted steel, structural studies were carried out only on the steels Nos. 1-6. These studies revealed that only steels Nos. 3 and 6 were completely austenitic. Steels Nos. 1,4 and 5 contained σ-phase, while steel No. 2 contained δ-ferrite. Fully austenitic steels 3 and 6 were tested in a first run for local corrosion resistance and mechanical strength. Table 2 show strength data for the two steels 3 and 6 tested and for a number of grades of nitrogen alloyed steel commercially available and/or reported in the literature.

TABLE 2

Material strength values at RT and Cr and Ni equivalents of a number of representative steel grades.

Steel No	Tensile yield limit $R_{p0.2}$ Nmm^{-2}	Ultimate strength R_m Nmm^{-2}	Rupture strain A 50 %	Hardness HV 30	Cr-eq 1) according to Schaeffler	Ni-eq 2)	Cr/Ni-eq
17	488	837	48.2	261	23.1	23.5	.98
18	502	807	43.1		21.11	20.6	1.02
19	609	895	38.1		24.0	25.7	.93
3	309	671	49.4	239	23.7	27	.88
6	390	827	51.4	287	25.8	33.22	.78
20	240	600	45	155	24.8	26	.95
21	565	778	33.0		26.86	12	2.24
22	320	700	50	175	26.7	24.6	1.08
23					21.10	18.9	1.12
26	300	600	30		23.5	22	1.07
27	590	750	30		28.5	18.9	1.51
33	432	785	49.7		26.05	26	1.0

1) Cr-eq.: % Cr + % Mo + 1.5 x % Si + 0.5 x % Nb

2) Ni-eq.: % Ni + 0.5 x (% Mn + % Cu + % Co) + 30 x (% C + % N)

To start with, the results of comparative studies of corrosion resistance of steels Nos. 3 and 6 and of commercial steels 17-23 will be presented below.

TABLE 3

Results of comparative corrosion testing

Steel No	Crevice corr. potential Esp mV/SCE	Critical temp. in Fe Cl_3, °C Pitting	Crevice corr.	Attack in a synthetic scrubber solution[1] Weight loss g/m^2 h
3	+ 65	40	< 23	10
6	> 785	65	45	0
17	+ 25			
18	- 70	< 23	< 23	10
19	+ 25	35	< 23	7
22		75	40	0
20	+ 105	45	< 23	8
21	+ 10 ± 25	35	< 30	7
23	- 30	< 23	< 23	11

[1] 11.4 % H_2SO_4 + 1.17 % HCl + 1 % $CuCl_2$ + 1 % $FeCl_3$

Crevice corrosion

Crevice corrosion potential, Esp

Steel grade No. 6 immediately proved to be highly superior to most stainless steels ever tested by the applicant. While the best result of the reference materials was an Esp of +105 mV, steel No. 6 had not been attacked at Esp = +785 mV. At that level, the test had to be discontinued, because the clamps holding the specimens were severely corroded (they were made of steel grade NU SS 904L). This means that the steel grade tested can be considered fully resistant to sea water at room temperature.

Steel No. 3 were not nearly as resistant as steel No. 6 and ended up somewhat below the result of steel No. 20, in spite of having equal content of chromium and nickel and a molybdenum content only 1.5 and a nitrogen content only 0.21% lower than that of steel No. 6.

Critical crevice corrosion temperature, CCT

At the comparative CCT test in $FeCl_3$, steel No. 6 was atacked only when the temperature reached 45°C. Next to the best value, 40°C, was reached by steel No. 22, the rest of the CCT values being below room temperature, possibly with exclusion of steel No. 21, which was resistant up to 30°C.

Critical pitting temperature, CPT

This test was also carried out in $FeCl_3$. Table 3 indicates that steel No. 22 had the highest CPT value, 75°C, followed by experimental steel No. 6, 65°C. In comparison with the resistance to crevice corrosion, experimental steel No. 6 is less resistant to pitting. This may be a result of the number of inclusions in small laboratory charges, which is greater than that of material made at production conditions. The slag situation may be more significant to pitting than to crevice corrosion.

Corrosion in a synthetic scrubber solution

Only steels Nos. 6 and 22 were unattacked at 50°C.

TABLE 4

Evaluation of corrosion after 6 months exposure in a paper pulp bleaching department.

Steel No	Weight loss g	Max. pit depth mm	Speed of corrosion g/m^2 h	Crevice attack under distance washer, depth mm
16	.50	0.6	.004	< 0.1
24	1.16	1.1	.011	< 0.1
25	1.50	1.0	.013	.12

In order to evaluate further the corrosion properties of the steel according to the invention a laboratory charge of 50 kg, sample No. 16, was produced, of the same nominal composition as steel No. 6. This material also was forgable and rollable without problems and was similarly highly corrosion resistant. The results of

a field trial, laboratory corrosion tests, and a first test for weldability will be reported below.

The field trial was carried out in the chlorodioxide stage of a paper mill bleachery. The samples, which were small pieces of steel plate, were placed standing up in one corner of the inlet box of the filter. Half of the plates were partially immersed in the pulp suspension, the upper half being in the gas phase. The environment in these filters is so corrosive that there has not been satisfactory solution to the materials problem up to now. Earlier exposures have shown that only titanium resists attack. Some typical environment parameters are the following:

Redox potential 650 mV/SCE
Amount of active chlorine in filtrate 7 mg/l
pH 3.8
Chloride 220 mg/l
Temperature 73°C

The samples were of the steel grade No. 16 according to the invention and the comparison materials 24 and 25. All samples had been ground and pickled in 10% HNO_3 + 1% HF, 10 min at 60°C, prior to exposure.

The results are presented in Table 4.

Steel No. 16 had the lowest weight loss in g/m^2h and the lowest pit depth, while steel No. 25 had the highest weight loss and greatest pit depth. The exposure confirmed the laboratory data presented above on the 2 kg samples of steel No. 6.

The corrosion test carried out in the laboratory of the corrosion properties of steel No. 16 are summarized in Table 5. The steel was MIG welded with an electrode of the type Avesta P12. Especially noteworthy is that there were no corrosion attacks, neither in the weld nor in the heat affected zone. There were no problems asso-

ciated with the welding itself. The steel could be welded with a very low heat input, 0.235 kJ/mm. The weld was of a high quality, smooth, without spatter or pores.

TABLE 5

Summary of corrosion test results for steel No. 16, both base metal and weld.

SAMPLE	Pitting potential in 5% NaCl pH 2.2 90°C	Critical temperature, °C				HUEY per 1-5	STREICHER 120 h	STRAUSS 24 h
		in 6% $FeCl_3$		in synthetic scrubber solution				
	mV/SCE	Pitting	Crevice corrosion			Weight loss g/m^2 h		
BASE METAL	181	75	40	60 (CPT) 65 70	0 0.34 1.4 section surfaces only	0.27		
MIG weld	194	65				0.39	0.38	0

In order to evaluate also the features of the steel of invention produced on a commercial scale basis, there was produced a five tons heat, steel No. 35, table 1, group 1. Three ingots were casted. The ingots were forged and hot rolled to plates with 5 and 12 mm thicknesses without problem. The surfaces after rolling were in good condition. Trials made showed that after hot or cold rolling, the steel could be heat treated by heating to 1100°C, followed by quenching in water so as to obtain a pure recrystalized austenitic structure without precipitates and with the desired corrosion properties. The heat treated 5 mm-plate also was cold rolled to 3 mm and 1 mm thicknesses without problem.

The quench annealed hot rolled plate showed the following mechanical properties:

TABLE 6

Yield str.		Tensile strength	Elongation	Impact toughness	Hardness
$Rp_{0.2}$	N/mm^2	Rm	A_5 %	Charpy V, 10x5mm	HB
398		782	48.8	RT: 65 $-185^{\circ}C$:43	168

As far as corrosion testing was concerned, specimens from both 5 and 12 mm plates were tested in various environments. Generally speaking, the results were consistent with those made on laboratory melts and reported above.

CLAIMS

1. Steel grade of austenitic type, extremely resistant particularly to pitting and crevice corrosion, easily weldable, and of high material strength, c h a r a c t e r i z e d in that it has the following chemical composition in percentages by weight:

max 0.03	C
0.1 - 2	Si
8 - 15	Mn
15 - 30	Cr
12 - 20	Ni
3.5 - 10	Mo
0.35- 0.55	N

the balance substantially consisting only of iron, impurities, and accessory elements in normal concentrations.

2. Steel grade according to claim 1, c h a r a c t e r i z e d in that its so called PRE value (= %Cr + 3.3 x %Mo + 16 x %N) is at least 41, preferably from 41 to 45.

3. Steel grade according to claim 1, c h a r a c t e r i z e d in that it contains 14-17 Ni.

4. Steel grade according to claim 1, c h a r a c t e r i z e d in that it contains 9-11 Mn.

5. Steel grade according to claim 1, c h a r a c t e r i z e d in that it contains 18-23 Cr.

6. Steel grade according to claim 1, c h a r a c t e r i z e d in that it contains 4-8 Mo.

7. Steel grade according to claim 1, c h a r a c t e r i z e d in that it contains 0.38-0.48 N.

8. Steel grade according to any one of the claims 1-7, c h a r a c -

t e r i z e d in that it contains no more than 0.02 C and a total of not more than 0.1 % of niobium, tantalum, titanium and zirconium.

9. Steel grade according to any one of the preceding claims, c h a r a c t e r i z e d in that its Cr equivalent (= %Cr + %Mo + 1.5 x %Si + 0.5 x %Nb) is at least 24, that its Ni equivalent (= %Ni + 0.5 x %Mn + 30 (%C + %N)) is at least 25, and that the quotient Cr eq./Ni eq. is less than or equal to 0.9, preferably less than or equal to 0.8.

10. Steel grade according to any one of the claims 1-9, c h a r a c t e r i z e d in that it has the following composition

max. 0.020	C
0.2 - 1.5	Si
9 - 11	Mn
max. 0.008	S, preferably max. 0.005 S
19 - 22	Cr
14 - 17	Ni
4 - 5	Mo
0.38 - 0.48	N

the balance being substantially only iron and unavoidable impurities.

European Patent Office

EUROPEAN SEARCH REPORT

0142015
Application number

EP 84 11 2048

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	FR-A-2 228 119 (NIPPON STEEL) * claims; page 5, table *	1,3-7, 10	C 22 C 38/58
Y	FR-A-2 229 776 (ARMCO) * claims *	1,3-7, 10	
Y	FR-A-1 466 926 (STAHLWERKE SÜDWESTFALEN) * claims *	1,3-7, 10	
A	DE-B-1 205 289 (PHOENIX-RHEINROHR) * column 1, lines 11-45 *	2	
A	DE-B-1 950 932 (BÖHLER)		**TECHNICAL FIELDS SEARCHED (Int. Cl.4)**
A,D	CORROSION-NACE, vol. 32, no. 7, July 1976, pages 274-276, Houston, Texas, US; R.J. BRIGHAM et al.: "Localized corrosion resistance of Mn-substituted austenitic stainless steels: Effect of molybdenum and chromium"		C 22 C

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	29-01-1985	OBERWALLENEY R.P.L.I

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
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